(19) Europäisches Patentamt
European Patent Offic
Office européen d s brev ts

(11) Publication number: **0 152 747**
A2

(12) EUROPEAN PATENT APPLICATION

(21) Application number: 85100226.1

(22) Date of filing: 11.01.85

(51) Int. Cl.⁴: H 01 J 49/04

(30) Priority: 17.02.84 US 581476

(43) Dete of publiication of application: 28.08.85
Bulletin 85/35

(84) Designated Contracting Stetes: AT BE CH DE FR GB IT LI LU NL SE

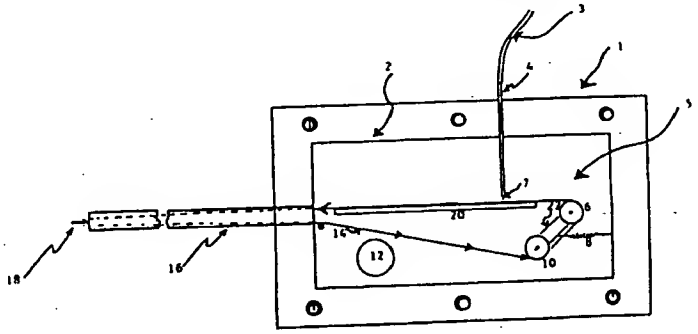
(71) Applicant: AMERICAN CYANAMID COMPANY,
1937 West Main Street P.O. Box 60, Stamford
Connecticut 06904 (US)

(72) inventor: Stout, Steven Jemes, 668 Paxon Avenue,
Mercerville New Jersey 08619 (US)
inventor: DaCunhe, Adrian, 4 Bliington Road, Hamilton
Squere New Jersey 08690 (US)

(74) Representative: Wächtershäuser, Günter, Dr., Tal 29,
D-8000 München 2 (DE)

(54) Apparatus and method for applying a dissolved sample from e ilquid chromatograph to e chemicel ionization mass spectrometer.

(57) The present invention relates to an apparatus thet deliv-ers e liquid chromatographic fractions sample directly into a chemical ionization mass spectrometer by utiiizing e single interface unit. This invention also relates to the method for so directly delivering said sample into said chemical ionization mass spectrometer.



EP 0 152 747 A2

consists of a set of machined slits and an evacuated chamber evacuated by its own mechanical vacuum pump. Due to th presence of machined slits and evacuated chambers, existing moving-belt interfaces are rather large and cumbersome and are mounted to the mass spectrometer on a separate flange.

SUMMARY OF THE INVENTION

The present invention alleviates these problems and contains a single-chambered interface unit into which eluate (sample) is applied. This not only eliminates unnecessary bulkiness but also eliminates the need for additional vacuum locks. This, in turn, further simplifies the interface unit because unnecessary hardware is no longer needed.

Furthermore, the interface unit of the present invention may be easily inserted into and removed from a chemical ionization mass spectrometer via the mass spectrometer's conventional solid probe inlet thereby alleviating the necessity of using special equipment in assembly and/or disassembly, as do conventional atmospheric moving belt interface units.

The present single-chambered moving-belt interface unit comprises a means for connecting the outlet of a liquid chromatograph, such that the liquid chromatographic eluate is deposited onto the moving belt of the unit. The apparatus of the present invention also contains a means for transferring (connecting and introducing) the liquid chromatographic sample, now dissolved, into the conventional chemical solid probe of the ionization chamber of a chemical ionization mass spectrometer.

In operation, standard connecting means, originating from the liquid chromatograph outlet, link the outlet from which a sample can be deposited onto the moving belt of the present interface unit.

The moving belt of the present apparatus is driven by some means, and this driving mechanism can be

mounted either externally or internally.

In addition to the previously-described elements of the present interface unit, the chamber that houses the moving belt should be evacuated by any suitable means.

It also may be necessary to assist in the evaporation of the liquid chromatograph sample deposited onto the moving belt. Any suitable heating system may be incorporated into the present invention to so evaporate the samples.

Once the sample (eluate) is deposited onto the moving belt of the present interface unit and is evaporated, it is ready to be introduced into the ionization chamber of the chemical ionization mass spectrometer. This is accomplished by direct connecting of an evacuated probe leading from the evacuated chamber of the present invention and located such that the evaporated sample is easily transported to any conventional solid probe of such mass spectrometers.

It is an object of the present invention, therefore, to provide an evacuated single-chambered moving belt interface unit for and a method of continuously applying the evaluate of a liquid chromatograph to a chemical ionization mass spectrometer through connection to the conventional solid probe inlet of said mass spectrometer. This and further objects will become apparent from the description of the drawing and detailed description of the preferred embodiment of the invention which follow.

BRIEF DESCRIPTION OF THE DRAWING

The figure shows a schematic diagram of one of the embodiments of the apparatus of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring to the figure, there is illustrated a liquid-chromatograph-chemical-ionization-mass-spectrometer interface unit, 1. The unit includes a vacuum housing, 2, having some means for maintaining the vacuum environment of

the unit. In the figure, a vacuum port, 12, connects a vacuum pump (not shown) to the housing, 2.

The eluate from the chromatograph, 7, is transported from the liquid chromatograph outlet via liquid chromatographic tubing, 3, linked to a capillary applicator, 4, passing through the vacuum housing into the interior chamber, 5, of the present interface unit.

The eluate is then deposited onto a continuous (moving) belt, 14. This internally-located moving belt, 14, is driven by some driving means. In the embodiment shown in the figure, an idler/pulley system, 10, over which the moving belt, 14, loops, fulfills this function of moving the belt because the idler/pulley system connects to a drive shaft (not shown) mounted onto a mounting block, 22, that passes through the interior chamber, 5, and vacuum housing, 2, to an external electric motor (not shown). This electric motor provides the drive to rotate several guides, rollers and pulleys, not shown, in moving the belt. Furthermore, an extension spring, 8, connecting the drive pulley, 6, of the idler/pulley system through these guides and rollers, maintains that system under tension for the belt to move properly.

Additionally, it may be necessary to evaporate the deposited eluate. This is accomplished by a heater, 20, located within the inner chamber of the present invention in such a manner as to be located beneath the moving belt and at the point where eluate is deposited onto said moving belt.

Once the eluate is deposited and evaporated, it moves along with the moving belt into an evacuated probe, 16, and into the solid probe tip (notched probe tip), 18, of the ionization chamber of a mass spectrometer. The eluate is deposited into the solid probe, 18, and the moving belt, 14, then moves back into the evacuated probe, 16, and into the inner chamber, 5, of the evacuated housing, 2. It is guided with rollers and such, not shown, along the way.

The method of the present invention now permits the continuous introduction of a solution (sample), 7, at

flow rates of up to 200 μL/minute of methanol, into a chemical ionization mass spectrometer. For instance, a sample, 7, originating from chromatograph, not shown, and transported through chromatographic tubing, 3, to capillary application, 4, is deposited onto moving belt, 14, of the present interface unit. This unit is under reduced pressure.

Then, sample, 7, is evaporated by heater, 20, located directly beneath capillary applicator,4, and moving belt, 14, and the sample, 7, is then transferred via the moving belt, 14, to an evacuated probe, 16, connected to a notched probe tip, 18, leading to the ionization chamber of the chemical ionization mass spectrometer (not shown).

The moving belt then continues back through evacuated probe, 16, to housing, 2, around the idler/pulley system, 10, to again move a sample through the unit.

Examples of the introduction of samples into a chemical ionization mass spectrometer under a variety of conditions utilizing the interface unit of the present invention are further illustrated in Table 1.

TABLE I

Operating pressures and principal ions

CH_3OH Flow Rate (μL/min)	Source Pressure (torr)	Interface Pressure (Torr)	Principal Ions (m/z)
0	0.40 (CH_4)	0.26	17^+ (CH_5^+)
			29^+ ($C_2H_5^+$)
			41^+ ($C_3H_5^+$)
100	0.25	0.35	65^+ $(CH_3OH)_2H^+$
			97^+ $(CH_3OH)_3H^+$
200	0.40	0.70	
100	0.40[a]	-	33^+ $(CH_3OH)H^+$
			57^+ ($C_4H_9^+$)
100	0.45[a]	-	57^+ ($C_4H_9^+$)

[a]Balance from isobutane

0152747

WHAT IS CLAIMED IS:

1. An evacuated single-chambered moving-belt interface unit connecting the outlet of a liquid chromatograph with a conventional solid probe of the ionization chamber of a chemical ionization mass spectrometer, said interface unit comprising: means for connecting said outlet of said liquid chromatograph to said interface unit; means for depositing a sample originating from said liquid chromatograph outlet onto said moving belt located within an evacuated housing; means for driving said moving belt; and means for transferring said sample to said conventional solid probe of said ionization chamber.

2. An evacuated single-chambered moving-belt interface unit according to Claim 1 additionally comprising: means for evacuating said unit's single chamber.

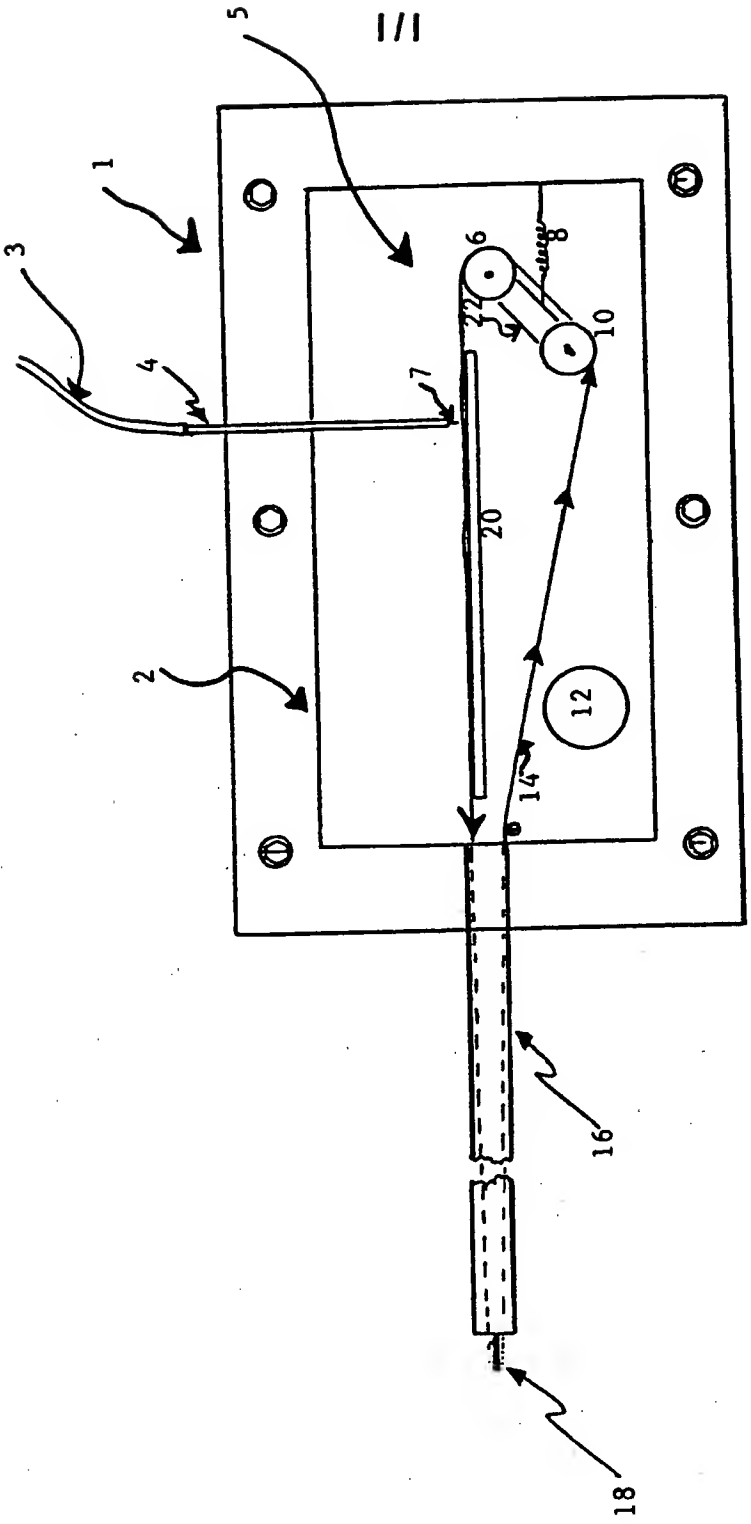
3. An evacuated single-chambered moving-belt interface unit according to Claim 2 additionally comprising: means for heating and thereby evaporating said sample.

4. An evacuated single-chambered moving-belt interface unit connecting the outlet of a liquid chromatograph with a coventional solid probe of the ionization chamber of a chemical ionization mass spectrometer, said interface unit comprising: an evacuated chamber housing a moving belt, said moving belt driven by an idler/pulley system connected to an externally-located electric motor; a capillary applicator having one end connected to chromatographic tubing leading from said outlet of said liquid chromatograph and having the other end positioned in said chamber, such that sample from said liquid chromatograph is deposited on said moving belt; a heater located beneath said moving belt and at the point said sample is deposited onto said moving belt; and an evacuated probe connected to said evacuated chamber and leading to said conventional solid probe of said mass spectrometer; wherein said moving belt moves in such a fashion as to transfer said sample from said evacuated chamber to said evacuated probe and to said conventional solid probe and then returns again to said

evacuated chamber.

5. An evacuated single-chambered moving-belt interface unit according to Claim 4, wherein said conventional solid probe is a notched probe tip of said ionization chamber of said chemical ionization mass spectrometer.

6. A method for directly introducing a liquid chromatograph sample into a chemical ionization mass spectrometer, said method comprising: introducing said sample into an evacuated single-chambered moving-belt interface unit, under reduced pressure; depositing said sample onto a moving belt located in said chamber; evaporating said sample; and transferring said sample to the notched probe tip of the ionization chamber of said chemical ionization mass spectrometer.


5
1
3
4
6
8
22
10
7
20
2
12
14
16
18